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NiO/YSZ Reduction for SOFC/SOEC Studied In Situ by Environmental Transmission Electron Microscopy

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SOFCs/SOECs are typically composed of ceramic materials, which are highly complex at the nano-scale. Scanning and transmission electron microscopy (SEM and TEM) are routinely applied for studying these nano-scaled structures post mortem, but only few SOFC/SOEC studies have applied environmental TEM (ETEM). ETEM offer the possibility to record image series (movies) of the ceramic nanostructures with atomic scale resolution during exposure to a reactive gas environment at elevated temperatures. The present contribution focuses on the typical reduction preparation step for the state-of-the-art Ni/YSZ (YSZ = Y_2O_3-stabilized ZrO_2) based anodes for SOFC and cathodes for SOEC. Specifically, the reduction of nickel oxide to form the catalytically active nickel surface is monitored directly at the nano- and atomic scale by using an ETEM. The reduction process was followed while exposing NiO/YSZ and pure NiO to H_2 at temperatures from room temperature to ca. 800°C. The NiO/YSZ was prepared by crushing down a tape casted and sintered model anode/cathode into a fine powder. Previous studies based on averaging techniques have shown that the reduction of pure NiO is a relatively rapid process, while the reduction of NiO/YSZ is slower, which indicates that the presence of YSZ inhibits the reduction of NiO. In the presents in situ experiments the temperature dependent reduction profile are found similar for the both nano-scaled NiO and NiO/YSZ sample. The apparent inhibitive effect of YSZ on NiO reduction is therefore not caused by a direct interaction between NiO and YSZ, but is an indirect effect depending on the NiO being integrated in a macroscopic network of NiO/YSZ. A Titan E-Cell 80-300ST TEM was used for the in situ work in combination with the chip-based heating holder from Protochips which facilitated rapid temperature ramping for example from room temperature to 800°C in only 1 s. The ETEM results are compared to complementary averaging techniques such as thermo-gravimetric analysis (TGA) and X-ray diffraction analysis (XRD).

The figure presents a TEM image series of NiO during exposure to 2 mbar H_2 and constant temperature ramping rate of 1°C/min. The NiO observed in the first image at 320°C is dense. From

the lower left corner a front of porous Ni is progressing until full reduction at 340°C.